

INTERNATIONAL STANDARD



2829

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Aluminium oxide primarily used for the production of aluminium — Determination of phosphorus content — Reduced phosphomolybdate spectrophotometric method

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FOREWORD

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Draft International Standards adopted by the Technical Committees are circulated to the Member Bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 2829 was drawn up by Technical Committee ISO/TC 47, *Chemistry*, and circulated to the Member Bodies in June 1972.

It has been approved by the Member Bodies of the following countries :

Australia	India	South Africa, Rep. of
Austria	Ireland	Sweden
Belgium	Italy	Switzerland
Canada	Morocco	Thailand
Czechoslovakia	Netherlands	Turkey
France	Poland	United Kingdom
Germany	Portugal	U.S.S.R.
Hungary	Romania	

This International Standard has also been approved by the International Union of Pure and Applied Chemistry (IUPAC).

The Member Body of the following country expressed disapproval of the document on technical grounds :

New Zealand

Aluminium oxide primarily used for the production of aluminium – Determination of phosphorus content – Reduced phosphomolybdate spectrophotometric method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a spectrophotometric method using reduced ammonium phosphomolybdate for the determination of the phosphorus content of aluminium oxide primarily used for the production of aluminium.

The method is applicable to phosphorus contents, expressed as P_2O_5 , greater than 0,000 5 %.

2 REFERENCE

ISO/R 804, *Aluminium oxide primarily used for the production of aluminium – Preparation of sample solution for analysis*.

3 PRINCIPLE

Preparation of a solution from a test portion by alkaline fusion, either with a mixture of sodium carbonate and boric acid, or with a mixture of sodium carbonate and sodium tetraborate. Dissolution of the melt in nitric acid and adjustment of an appropriate aliquot portion to pH 2.

Formation of the phosphomolybdic complex and extraction by 2-methylpropan-1-ol in a sulphuric acid medium.

Reduction of the complex by tin(II) chloride in the organic phase and spectrophotometric measurement of the reduced complex contained in the organic phase at a wavelength of about 730 nm.

4 REAGENTS

Distilled water, or water of equivalent purity, shall be used in the test.

4.1 Sodium carbonate, anhydrous.

4.2 Boric acid (H_3BO_3), or alternatively

4.2.1 Sodium tetraborate, anhydrous ($Na_2B_4O_7$).

4.3 2-Methylpropan-1-ol (*isobutyl alcohol*), ρ approximately 0,805 g/ml.

4.4 Nitric acid, approximately 8 N solution.

Dilute 540 ml of nitric acid solution, ρ approximately 1,40 g/ml, about 68 % (*m/m*) solution, with water to 1 000 ml.

4.5 Sulphuric acid, approximately 10 N solution.

Carefully add, in small portions, 280 ml of sulphuric acid, ρ approximately 1,84 g/ml, about 96 % (*m/m*) solution, to about 500 ml of water and, after cooling, dilute to 1 000 ml.

4.6 Sulphuric acid, approximately N solution.

Take 100 ml of the sulphuric acid solution (4.5) and dilute with water to 1 000 ml.

4.7 Sulphuric acid, approximately 0,5 N solution.

Take 250 ml of the sulphuric acid solution (4.6) and dilute with water to 500 ml.

4.8 Ammonium acetate, 500 g/l solution.

4.9 Iron(III) sulphate, acid solution.

Dissolve 0,5 g of iron(III) sulphate nonahydrate [$Fe_2(SO_4)_3 \cdot 9H_2O$] in 50 ml of water containing 2 ml of perchloric acid solution, ρ approximately 1,60 g/ml, about 64,5 % (*m/m*) solution, and dilute to 100 ml.

1 ml of this solution contains about 0,001 g of Fe(III).

NOTE — If iron(III) sulphate nonahydrate of recognized analytical reagent quality is not available, ammonium iron(III) sulphate dodecahydrate may be used. In this case, to obtain a solution with the same concentration of iron(III), dissolve, instead of 0,5 g of iron(III) sulphate nonahydrate, 0,86 g of ammonium iron(III) sulphate dodecahydrate [$FeNH_4(SO_4)_2 \cdot 12H_2O$].

4.10 Ammonium iron(II) sulphate, acid solution.

Dissolve 0,5 g of ammonium iron(II) sulphate hexahydrate [$Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$] in 50 ml of water containing 0,5 ml of perchloric acid solution, ρ approximately 1,60 g/ml, about 64,5 % (*m/m*) solution, and dilute to 100 ml.

1 ml of this solution contains about 0,000 7 g of Fe(II).

Prepare this solution just before use.

4.11 Ammonium molybdate, 25 g/l acid solution.

Dissolve 5 g of ammonium molybdate tetrahydrate [$(NH_4)_6Mo_7O_{24} \cdot 4H_2O$] in water at 60 °C. Cool and dilute to 100 ml. Add to the solution 100 ml of the sulphuric acid solution (4.5) and mix.

Store the solution in a plastics bottle.